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AN ANALYTICAL METHOD FOR DETECTING BLOWN-OUT SHOTS IN COAL MINES

BY

G. F. HUTCHISON and JACOB BARAB

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AN ANALYTICAL METHOD FOR DETECTING BLOWN-OUT SHOTS IN COAL MINES.

By G. F. Hutchison and Jacob Barab.

INTRODUCTION.

A blown-out shot is the term applied by coal miners to a shot in which the explosive brings down no coal, the main force being expended on the air of the mine. Such shots result from improper placing or charging of the bore hole and are most common in shooting off the solid. They are dangerous and are to be avoided.[a] The danger lies in the possible ignition of fire damp or dust particles by the flame that shoots from the bore hole. Blown-out shots have caused many serious explosions in gaseous or dusty mines.

In the investigation of coal-mine explosions it is often important to ascertain whether there was a blown-out shot and if so whether black powder or a high explosive was used. To determine this, samples scraped from bore holes and from the sides of powder boxes have frequently been submitted to the explosives laboratory of the Bureau of Mines by the mining engineers in their study of mine explosions to be analyzed for solid products of combustion of explosives. Because of the scanty knowledge then at hand it was impossible to determine by any simple method of analysis whether the soluble matter found in the coal came from an explosive or to interpret satisfactorily the results obtained. Therefore an investigation of the solid products obtained from bore holes from both black powder and permissible explosives under definite known conditions approximating those in mines was made at the explosives chemical laboratory of the Bureau of Mines in Pittsburgh, Pa., in order to devise, if possible, a simple method of analyzing the solid products of explosions so as to obtain the desired information. This paper presents an account of the investigation.

ACKNOWLEDGMENTS.

Acknowledgment is due to W. C. Cope, former explosives chemist of the Bureau of Mines, who originally advised the under-taking of this study; to S. P. Howell, explosives engineer, who made valuable suggestions in outlining the tests; and to E. H. Denny, mining engineer, A. J. Strane, assistant explosives engineer, and H. C. Howarth, mine superintendent, who supervised the placing and firing of the shots at the mine.

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[a] Rutledge, J. J., Use and misuse of explosives in coal mining: Miners' Circular 7, Bureau of Mines, 1913, 51 pp.

PROCEDURE.

EXPLOSIVES USED.

The solid products of combustion obtained would depend on the type of explosive used, the method of priming, and the manner of charging the hole, that is, with or without stemming.

Six different explosives were used in the first set of tests—one a black powder, the other five selected from each class of the explosives on the permissible list.[a] The analysis of the black blasting powder (B–2078) is as follows:

Analysis of B-2078, black blasting powder.

	Per cent.
Moisture	0.3
Sodium nitrate	72.5
Sulphur	12.0
Charcoal	15.2
	100.0

As the Bureau of Mines does not publish the analysis of permissible explosives, only the approximate composition of these can be given.

D–2050 is an explosive of class 1a. It contains ammonium nitrate and sodium nitrate in the proportion of about 8 to 1. Nitroglycerin, carbonaceous combustible material, and calcium carbonate are also present.

M–2061 is an explosive of class 1b. Its chief ingredients are ammonium nitrate and sodium chloride in the proportion of about 7 to 1. Trinitrotoluene and small porportions of zinc oxide and carbonaceous combustible material are also present.

D–2062 is an explosive of class 2. It contains potassium nitrate, ammonium sulphate, and ammonia alum in about equal proportions, also nitroglycerin, carbonaceous combustible material, and clay.

M–2028, an explosive of class 3, contains about 32 per cent of ammonium nitrate, 8 per cent of sodium nitrate and 28 per cent of dolomite. The content of carbonaceous combustible material is low.

D–1972, an explosive of class 4, has more carbonaceous combustible material than any of the other permissible explosives used in these shots. It also contains nitroglycerin and sodium nitrate.

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[a] For the classification of permissible explosives, see Fay, A. H., Production of explosives in 1918, Tech. Paper 231, Bureau of Mines, 1919, p. 15.

METHOD OF PRIMING.

Shots of black powder were primed with igniters, but in firing the permissibles one charge of each was primed with a No. 6 electric detonator and one with an igniter and half a gram of guncotton. The purpose of the latter method was to cause the explosive simply to burn, thus simulating the condition when too weak a detonator is used and an incomplete detonation results.

Stemming was not used in the shots primed with an igniter and guncotton. In all other charges stemming was used and it was well tamped. The shots and shot-firing arrangements were planned and made by explosives engineers and mining engineers of the bureau.

Twelve bore holes were used in the first set of tests. All the holes were drilled in the face of room 4, off the first butt, off the main air course at the experimental mine of the Bureau of Mines at Bruceton, Pa. The face of the room at the time of these shots was well squared up. All the holes were drilled into the solid without undercutting, so that no coal would be brought down and "blown-out" shots would result.

For the sake of uniformity it was thought best to take such a quantity of each explosive that each charge would have the same explosive strength. Accordingly, the quantity of powder used in each shot was the unit deflective charge for that particular powder with the exception of the black-powder shot in hole No. 12, in which twice that amount was fired.[a]

METHOD OF SAMPLING.

In drilling the holes all the coal drilled from two of them was kept for comparison with the scrapings from the holes after the shots.

After the shots were fired and the gas cleared out of the mine, all the loose material at the collar of each hole was removed as one sample. Then another sample was taken from the remainder of the hole and the hole scraped through its entire length. This method of taking two samples from each hole was followed only in the first set of tests. For reasons which will be set forth later it was thought advisable to combine all the scrapings from a single hole into one sample.

The details of firing and sampling are summarized in Table 1. The key numbers in the first column are those assigned to each sample of scrapings while those in the fourth column show which explosive was fired in each hole and refer to the descriptions already given.

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[a] The unit deflective charge is that weight of explosive which will produce a swing of the ballistic pendulum exactly equal to that effected by a half-pound charge of "standard" 40 per cent dynamite. See Hall, Clarence, Snelling, W. O., and Howell, S. P., Investigations of explosives used in coal mines: Bulletin 15, Bureau of Mines, 1912, p. 79.

TABLE 1.—*Key data and physical examination of sample.*

Key number.	From hole No.	Section of hole.	Powder fired in hole.	Device used for firing.	Stemming used, inches.	Sample taken, grams.	Through 8-mesh sieve, grams.	Examination of hole after firing and remarks.
1	2	3	4	5	6	7	8	9
2098	1	(a)	....	....	....	3,053	2,248	
2099	8	(a)	....	....	....	2,873	2,802	
2100	1	Collar	D-1972	E. D.[b]	10	83	40	Shot broke 3 inches off collar of hole and caused a vertical crack on face 1 foot above and 1 foot below.
2101	1	Back				430	233	Much fines—difficult to scrape.
2102	2	Collar	B-2078	Igniter	11	28	18	A part of the stemming found in place.
2103	2	Blocking hole 2 feet 6 inches from collar.				110	87	
2104	2	Back				111	28	
2105	3	Collar	D-1972	Igniter and guncotton.	None.	43	25	Charred paper found in back of hole.
2106	3	Back				114	71	
2107	4	Collar	M-2028	E. D.[b]	9	26	21	
2108	4	Back				273	231	Back of hole seemingly much shattered. Much fines and many small lumps found.
2109	5	Collar	M-2028	Igniter and guncotton.	None.	67	43	2 inches of explosive was found in bottom of hole with some charred paper.
2110	5	Back				330	200	
2111	6	Collar	D-2050	E. D.[b]	7	116	84	Back of hole was shattered, and produced considerable fines.
2112	6	Back				645	325	
2113	7	Collar	D-2050	Igniter and guncotton.	None.	146	39	5 inches of explosive was found in bottom of hole after shot.
2114	7	Back				86	63	Little material found in hole after shot, except unexploded powder.
2115	8	Collar	M-2061	E. D.[b]	7½	131	81	Back of hole shattered considerably and a crack extended to the face.
2116	8	Back				400	184	
2117	9	Collar	M-2061	Igniter and guncotton.	None.	31	25	After the shot explosive particles were observed along the bottom of the bore hole, but none could be found at the back of the bore hole.
2118	9	Back				93	59	
2119	10	Collar	D-2062	Igniter and guncotton.	None.	41	20	After shot, 1 foot of collar gone, and transverse cracks formed 1 foot from back and at back. Cracks probably due to shot in hole No. 12, next to it and fired at same time.
2120	10	Back				92	58	Hole after shot felt soft at back, possibly paper from cartridge or some explosive.
2121	11	Collar	D-2062	E. D.[b]	5¾	84	45	Back of hole considerably shattered by shot and 3 inches of collar blown off.
2122	11	Back				486	244	Large amount of fines in back portion of hole after shot.

[a] Entire drillings of hole.

[b] E. D.—electric detonator.

TABLE 1.—*Key data and physical examination of sample.*—Continued.

Key number.	From hole No.	Section of hole.	Powder fired in hole.	Device used for firing.	Stemming used, inches.	Sample taken, grams.	Through 8-mesh sieve, grams.	Examination of hole after firing and remarks.
1	2	3	4	5	6	7	8	9
2123	12	Scrapings from entire length of hole.	B-2078	Igniter......	9	251	57	About 2 feet blown off collar of hole. On the face, a ½-inch crack extended through the hole from the draw slate to 2 feet below hole. In order to get scrapings considerable coal had to be picked away from the collar to a point where the crack was not wide enough to catch too much of sample.

METHOD OF EXAMINATION AND ANALYSIS.

Each sample was removed from its container, weighed, and carefully observed with respect to paper, free powder, odor, and any other properties which could be noted. The sample was then sifted on an eight-mesh sieve. Large particles remaining on the sieve were well dusted with a camel's-hair brush, so that any powder residue adhering might pass through. The part that passed through the sieve was also weighed, and only this latter part was used for analysis.

Convenient portions of each sample up to 100 grams were weighed out, placed in 400-c.c. beakers, and covered with about 250 c.c. of cold distilled water. The samples were stirred well and allowed to stand for about one hour. Then they were filtered into 500-c.c. graduated flasks and the coal washed with cold distilled water until the flask was filled to the mark.

The solutions were tested with litmus paper to determine whether acid, alkaline, or neutral. Qualitative tests were made for sulphate, thiosulphate, sulphite, sulphide, carbonate, chloride, ammonia, nitrate, calcium, and cyanide. Where these tests indicated appreciable quantities, a quantitative determination was made by taking an aliquot part of the solution and proceeding in the usual manner.

Sulphate was determined gravimetrically by precipitation as barium sulphate, and sulphite and thiosulphate together were determined in like manner after oxidation with bromine water. No sulphide could be detected in any of these or the subsequent samples. Chlorine was determined gravimetrically as silver chloride, and cyanide, in the absence of chlorine, as silver cyanide. Carbonates were determined by titration with standard sulphuric acid and methyl orange indicator, ammonia by distillation into standard acid, nitrates either by precipitation with nitron or in the nitrometer.

TABLE 2.—*Analysis of coal samples.*

Method of priming.	Key numbers of samples.	Grams of sample extracted.	Powder used.	Number of hole and section.	Appearance of solution.	Analysis, in grams per 100 grams, of 8-mesh sample.							
						Evaporated residue.		SO$_3$.	CO$_2$.	NH$_4$.	NO$_3$.	Miscellaneous.	Reaction to litmus.
						Dried at 105° C.	Ignited.						
	[a] 2098	100	None	1	Clear	0.047	0.026	0.0113					Neutral.
	[a] 2099	100	None	8	do.	.03	.014	.0039					Do.
Igniter	2102	15	B-2078	2-collar	do.	.2	.127	.0228	Trace				Do.
	2103	50		2-middle	do.	.632	.566	.1173	do.				Alkaline.
	2104	25		2-back	Turbid	2.632	2.128	.2512	0.2825			0.293 SO$_2$	Do.
	2123	50		12-entire length	Clear	1.906	1.842	.1673	.3620			.4522 SO$_2$	Do.
Electric detonator	2100	35	D-1972	1-collar	do.	.248	.185	.0127	.0605				Do.
	2101	100		1-back	Turbid	.725	.604	.0161	Trace				Do.
	2107	20	M-2028	4-collar	do.	.580	.365	.1455					Neutral.
	2108	100		4-back	Clear	.148	.116	.0089	.388				Alkaline.
	2111	80	D-2050	6-collar	do.	.115	.086	.0184	.0221				Do.
	2112	100		6-back	do.	.126	.093	.0069	.0318				Do.
	2115	50	M-2061	8-collar	do.	.272	.096	.0192	.0212				Do.
	2116	100		8-back	do.	.175	.121	.0117	.0035				Do.
	2121	45	D-2062	11-collar	do.	.313	.267	.1242	.0313				Do.
	2122	100		11-back	do.	.262	.215	.1207	.0141				Do.
Igniter and guncotton	2105	25	D-1972	3-collar	Yellow	12.028	9.516	.0728	3.0084		Trace	.1616 CN	Do.
	2106	50		3-back	do.	20.886	19.674	.0124	7.5506		.7345	.3014 CN	Do.
	2109	25	M-2028	5-collar	do.	4.272	1.484	.1304	.226	0.5001	2.5227		Do.
	2110	100		5-back	Brown	4.093	3.591	.2661	1.0628	.0397	.4221		Do.
	2113	35	D-2050	7-collar	Clear	.714	.174	.0088	.0403	.1118	.5147		Do.
	2114	60		7-back	do.	2.7	.298	.0057	.0353	.5120	2.1585		Do.
	2117	25	M-2061	9-collar	Yellow	36.96	5.416	.0603	.1977	6.1798	Present	3.6508 Cl	Do.
	2118	50		9-back	do.	23.352	4.026	.0399	.1059	3.3771	do.	2.1382 Cl	Do.
	2119	15	D-2062	10-collar	Clear	1.033	.773	.2606	.1177	.0276			Do.
	2120	50		10-back	do.	7.942	3.758	3.0553	Trace	.9174	1.8 808	.1642 Al$_2$O$_3$	Acid.

[a] Coal drillings.

In addition to these determinations the total water-soluble constituents of each solution was determined by evaporating 100 c.c. to dryness in a platinum dish on a water bath and drying for about three hours in an oven at 105° C. After cooling and weighing, the dish and contents were ignited to incipient redness over a free flame, cooled in a desiccator, and again weighed. All results were calculated on a percentage basis.

In many samples the manner in which the basic and acid radicals were combined was uncertain; for instance, whether nitrate was present as sodium or ammonium nitrate. Therefore sulphate is expressed as SO_3, sulphite and thiosulphite (together) as SO_2, etc., as seen in the tables.

The analytical results are shown in Table 2, grouped according to the method of firing, as general conclusions regarding this first lot of samples will be drawn.

OBSERVATIONS ON TABLE 2.

1. Appreciable quantities of sulphates are to be expected in the residues of the holes from the black-powder shots. Except for the sample from the collar of the hole, the results sustain this expectation. The amount of evaporated residue should be fairly high and with the same exception this is likewise true. Both of these values should be considerably higher than for the residue from the detonating explosives, which, in general, is also true.

2. The detonating explosives primed with an igniter and guncotton did not explode completely; consequently they left considerable residue and should yield enough of the original powder or residues from it to enable one to identify the type of powder used.

To some extent the above observations could be made by careful inspection of the table. However, the analysis of the samples from the different parts of the same hole, instead of throwing light on the matter, seemed to confuse the results so that definite conclusions were difficult to draw. On further consideration, therefore, the calculation of the results on the basis of an average sample from the entire hole seemed advisable.

A specific example will best illustrate the method of making these calculations. Average sample from hole No. 3 included 2105 and 2106.

2105 contained 25 grams which passed through 8-mesh sieve.
2106 do 71 grams do do do do do
Total, 96 grams do do do do do
2105 contained 12.028 grams evaporated residue dried at 105°.
2106 do 20.886 grams do do do do do

$$\frac{25}{96}\times 12.028 = 3.194 \text{ grams}$$

$$\frac{71}{96}\times 20.886 = \underline{15.449} \text{ grams}$$

18.643 grams=evaporated residue per 100 grams of average sample from entire hole.

Table 3 contains results of analysis based on an average sample of 100 grams for the entire hole.

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TABLE 3.—*Results of Table 2 calculated on the basis of entire sample from each hole.*

Method of priming.	Hole number.	Key numbers assigned to samples.	Powder used.	Analysis, in grams per 100 grams of sample.						
				Evaporated residue.		SO_3	CO_2	NH_4	NO_3	Miscellaneous.
				Dried at 105°.	Ignited.					
	1	[a] 2098	....	0.047	0.026	0.0113	....	....	....	
	8	[a] 2099	....	.03	.014	.0039	....	....	....	
Igniter	2	2102, 2103, and 2104	B-2078	.995	.835	.1326	0.0595	....	....	0.0617 SO_2.
	12	2123	B-2078	1.906	1.842	.1673	.362	....	....	.4522 SO_2.
Electric detonator	1	2100 and 2101	D-1972	.66	.546	.0156	.0081	....	....	
	4	2107 and 2108	M-2028	.184	.136	.0203	.0356	....	....	
	6	2111 and 2112	D-2050	.128	.092	.0093	.0297	....	....	
	8	2115 and 2116	M-2061	.205	.113	.0137	.0089	....	....	
	11	2121 and 2122	D-2062	.27	.223	.1214	.0167	....	....	
Igniter and guncotton	3	2105 and 2106	D-1972	18.643	18.055	.0281	6.3677	....	0.5432	.265 CN.
	5	2109 and 2110	M-2028	4.125	3.224	.2432	.916	0.1214	.795	
	7	2113 and 2114	D-2050	1.943	.251	.0069	.0372	.3589	1.53	
	9	2117 and 2118	M-2061	27.40	4.453	.0461	.1334	4.214	Present.[b]	2.575 Cl.
	10	2119 and 2120	D-2062	6.176	2.99	2.3386	.03	.6892	1.40	.122 Al_2O_3

[a] Coal drillings.

[b] Present in appreciable quantity. Not determined.

OBSERVATIONS ON TABLE 3.

1. The question as to whether any explosive has been fired in a drill hole can be answered at once by means of the total water-soluble content. All scrapings show proportions of water-soluble matter appreciably greater than those of the coal samples.

2. The two holes in which black powder was fired gave scrapings higher in sulphate and salts of the lower acids of sulphur than the detonated shots and are thus distinguished from them. One of the detonated shots (hole No. 11) gave nearly as much sulphate as the black powder shot in hole No. 2. However, the ingredients of this powder (D–2062, p. 4) included about 33 per cent of ammonium sulphate and ammonia alum.

No sulphide could be detected in any of the samples from holes No. 2 and No. 12 or other subsequent samples containing black-powder residue, though this is generally regarded as one of the chief products of combustion of black powder. Sulphur of lower oxidation than the sulphate was found, however, and consisted of thiosulphate and sulphite. As no attempt was made to determine these separately in most of the samples, the two were reported together as SO_2. In one sample a quantitative determination of each was made and it was found that about one-fourth of the SO_2 came from sulphite and three-fourths from thiosulphate. Nobel and Abel,[a] who analyzed the solid products of combustion of black powder from a great many shots under varying pressures, found that in all samples thiosulphate was present. In shots fired under high pressure they found thiosulphate low and sulphide high. Low pressure gave residues high in thiosulphate and low in sulphide and a few residues containing no sulphide at all. The sulphate content of the residues varied little with the pressure.

The work of Nobel and Abel is mentioned to show that the absence of sulphides in the solid products of explosion of black powder has been observed before. Nobel and Abel worked with potassium nitrate powders containing 73.55 to 75.30 per cent potassium nitrate. As modern blasting powder contains about the same proportion of sodium nitrate, which has about 9 per cent more oxygen than potassium nitrate, the authors believe this condition is easily accounted for. They are unable to cite any extensive work on the solid products of combustion of sodium nitrate powders.

The thiosulphate reported by Nobel and Abel probably contained some sulphite as they determined it by simply titrating with iodine. As noted above these two salts were determined separately in one of the samples in the tests described. The method depends on their different reactions with iodine as seen from the following equations:

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[a] Nobel, Andrew, and Abel, F. A., Researches on explosives, fired gunpowder: Philos. Trans. Roy. Soc., vol. 165, 1875, pp. 49–155.

$$2\ Na_2S_2O_3 + I_2 = 2\ Na\ I + Na_2S_4O_6$$
$$Na_2SO_3 + I_2 + H_2O = Na_2SO_4 + 2\ HI$$

An aliquot part of the solution was first neutralized and then titrated with iodine which oxidized both the sulphite and thiosulphate. Then the hydriodic acid resulting from the action of the iodine on the sulphite was titrated with standard alkali after the addition of a few drops of phenolphthalein solution.

The presence of salts of lower oxidation than sulphate is important in distinguishing holes in which black powder was fired from those in which a permissible explosive containing a sulphate was fired (for example, hole 2 from hole 11, Table 3). No sulphite or thiosulphate has been found in residues from permissible explosives containing sulphate and it is not believed that such a reduction would take place. As sulphite and thiosulphate are distinguishing characteristics of the residues from black powder both in Table 3 and in Table 4 following, the presence of these salts proves conclusively that the residue is from black powder.

3. In general the total water-soluble matter from the detonated shots is much lower than that from the black-powder shots. In the single sample (hole No. 1), the evaporated residue of which approaches closely that of the lowest black powder (hole No. 2), the solution was somewhat turbid, probably caused by clay used as stemming. The evaporated residues also serve to distinguish this group from the first containing no residues from explosives, because the lowest residue of the former is about three times that of sample 2098.

4. The incompletely detonated shots can be identified at once by the presence of larger amounts of water-soluble matter than any of the preceding groups, owing to the fact that the solid products of combustion were not scattered by the shot, and by the presence of nitrate and, where ammonia dynamites were used, appreciable quantities of ammonia, both of which would be completely decomposed by detonation.

Further examination of these figures and comparison with the powders used discloses some interesting facts.

(*a*) Scrapings from hole No. 3 contained a large quantity of water-soluble matter, mostly sodium carbonate which lost very little weight on ignition. There being no ammonia, this can only correspond to D–1972, which has a large per cent of carbonaceous combustible material.

(*b*) Scrapings from hole No. 5 gave much water-soluble matter and contained ammonia and nitrate, together with a fair amount of carbonate. Of the four powders containing ammonium nitrate M–2028 is the only one to which this could correspond. It has low sulphate and no chloride; this eliminates M–2061 and D–2062. The loss on ignition is only one-fifth the total weight of the water-soluble, hence it could not be D–2050, 76 per cent of which is ammonium nitrate.

(*c*) Scrapings from hole No. 7, on the other hand, had an evaporated residue which lost about four-fifths of its weight on ignition. This fact, together with absence of chloride, low sulphate, and low carbonate, makes it correspond with D–2050 and no other.

(*d*) The residue of the scrapings from hole No. 9 contains enough chloride to establish that M–2061 was burned in it, which is further confirmed by the presence of some ammonia and the ratio of the loss on ignition to the total evaporated residue.

(*e*) The considerable sulphate and ammonia content of the residue of the scrapings from hole No. 11 clearly identifies D–2062 as the powder fired.

The conclusion from these observations was that by this method the following determinations could be made:

1. Whether or not a blown-out shot had occurred.
2. Whether the explosive detonated completely or not.
3. The nature of the explosive used if an incomplete detonation occurred.

By this last statement it is not meant that any explosive on the permissible list could be identified by examining its products of combustion. But of two or three explosives known to have been used in a certain mine, the analysis of the scrapings should generally tell which had been fired if the detonation was not complete.

TESTS OF THE METHOD.

Accordingly a number of unknown samples were submitted to try out the practicability of the method. The only information regarding these samples was that each one represented the entire scrapings from one hole and that the explosives that might have been used were black blasting powder, D–2050, D–2058, D–2064 and D–2215. Also two samples (assigned the numbers 2142 and 2230) were known to be nothing but coal and were to be used in comparison.

Below are descriptions of permissible explosives not previously given:

D–2058 has a large percentage of carbonaceous combustible material, and is almost identical with D–1972, previously described.

D–2064 resembles M–2061 except that it contains nitroglycerin instead of trinitrotoluene. This difference would not affect the solid products of combustion.

D–2215 contains ammonium nitrate, sodium nitrate, and gypsum.

Seventeen samples were submitted and analyzed. The analyses are given in Table 4.

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TABLE 4.—*Analysis of unknown samples.*

Sample No.	Total sample, grams.	Grams through 8-mesh.	Grams extracted.	Appearance of solution.	Action on litmus.	Analysis, in grams per 100 grams of sample.							
						Evaporated residue.		SO_3.	SO_2.	CO_2.	NH_4.	NO_3.	Miscellaneous.
						Dried at 105°.	Ignited.						
2139	196	95	90	Clear	Alkaline	1.082	1.041	0.39	0.2782	0.2496	....	....	
2141	313	103	100	do.	do.	1.075	1.037	.0285	.2867	.2434	....	....	
2142	241	173	100	do.	Neutral	.026	.006	....	....	.0094	....	....	
2144	561	287	100	Turbid	Alkaline	1.265	1.228	.0864	.3084	.2827	....	....	
2145	490	295	100	do.	do.	1.579	1.542	.1166	.5084	.2884	....	....	
2146	321	235	100	Clear	Neutral	.021	.003	....	....	.0075	....	....	
2147	475	262	100	do.	do.	.153	.0988	.0045	....	.0324	....	....	
2148	50	41	40	do.	do.	3.565	.5	.0154	....	.0368	0.7121	2.1685	0.309 Cl.
2150	277	176	100	Yellow	Alkaline	12.075	11.129	.0333	....	3.5348	....	Present[a]	.2074 CN.
2151	573	334	100	Clear	Neutral	.245	.126	.0038	....	.0112	....	....	.1004 Cl.
2152	179	64	60	do.	Alkaline	.968	.896	.0737	.1842	.2028	....	....	
2153	394	217	100	Yellow	do.	.83	.592	.0051	....	.1835	....	.0221	
2230	1,014	755	100	Clear	Neutral	.027	.016	.0017	....	.0075	....	....	
2231	175	39	35	do.	do.	.111	.077	.1018	....	.0268	....	....	.0171 CaO.
2232	653	154	100	do.	do.	.107	.081	.0237	....	.015	....	....	.0140 CaO.
2233	116	36	35	do.	Alkaline	.689	.637	.0852	.1438	.1337	....	....	
2334	117	79	75	do.	do.	3.798	2.596	.1879	.871	.525	....	....	

[a] Appreciable quantity but not determined.

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OBSERVATIONS ON TABLE 4.

1. From a study of the analyses in this table the following samples were reported to contain solid products of explosion of black blasting powder: 2139, 2141, 2144, 2145, 2152, 2233, and 2234. This conclusion was reached, first, because they all contained sulphite and thiosulphate (calculated as SO_2), and, second, because the other water-soluble salts were of the same character (sulphate and carbonate) and in about the same quantity as the known black powder residues in Table 3, holes No. 2 and No. 12.

2. Samples 2142 and 2230 were the coal samples known to be free from explosive residues and from its analysis 2146 was concluded to be the same.

3. Samples 2147, 2151, 2231, and 2232 all have a similar analysis. Sulphite (or thiosulphate) nitrate and ammonia are absent, which indicates that the water-soluble matter extracted did not come from the explosion of black powder or of an incompletely detonated high explosive. The amount of the soluble matter shows that the samples are not coal drillings, as the soluble matter in all four samples is several times that obtained from 2142, the coal sample. The same is true of the carbonate content. These residues were therefore reported to have come from drill holes in which a high explosive completely detonated.

4. Samples 2148, 2150, and 2153 contain much water-soluble material. This could not have come from black powder, for sulphites are absent and the sulphate content is low. The presence of appreciable quantities of nitrate in all three of these samples establishes the fact of incomplete detonation.

D–2050, D–2058, D–2064, and D–2215 might have been used to fire these holes, and reference was made to the description of these powders given above (p. 13), to determine which powder was used in each firing.

From the large quantity of chloride and ammonia present, 2148 must have come from a hole in which the powder D–2064 was incompletely detonated.

The powder D–2058 was assigned to sample 2150 because no ammonia was found in the water extract. Burning of a powder like D–2058 would also give a high carbonate and some cyanide, which was true of this sample. Of the known samples, that from hole No. 3 came from this type of permissible explosive and gave a similar analysis.

Absence of ammonia also indicated that burning of the same powder, D–2058, had produced the residue found in 2153. The constituents found are the same, although in much smaller proportion. This discrepancy could not be accounted for except by some irregularity in sampling or by a partial detonation blowing away some of the products.

When these samples had been reported the key cards describing the methods of charging and firing the shots were received for comparison. In every case except one (2153) the report made was correct, as will be seen from the data tabulated in Table 5.

TABLE 5.—*Key card data on unknown samples.*

Key number.	Powder fired.	Method of firing.	Stemming used.	Remarks.
2139	B-2078	Igniter	2 cartridges shale dust	
2140	None.			Sent to fuels laboratory.
2141	B-2078	Igniter	2 cartridges shale dust	
2142	None.			Official coal sample.
2143	B-2078	Igniter	2 cartridges shale dust	Sent to fuels laboratory.
2144	B-2078	do	do	
2145	B-2078	do	do	
2146	None.			
2147	D-2050	Electric detonator	2 cartridges shale dust	
2148	D-2064	Igniter and guncotton	None	Explosive found in hole.
2149	D-2050	Electric detonator	2 cartridges shale dust	Sent to fuels laboratory.
2150	D-2058	Igniter and guncotton	None	
2151	D-2064	Electric detonator	2 cartridges shale dust	
2152	B-2078	Igniter	do	
2153	D-2058	Electric detonator	do	
2230	None.			Official coal sample.
2231	D-2215	Electric detonator	10 inches of shale dust	
2232	D-2215	do	do	Blew off 8 inches of collar.
2233	B-2078	Igniter	do	Blew off 10 inches of collar.
2234	B-2078	do	do	

Sample 2153 was reported to be from powder D-2058, incomplete detonation. The powder in this hole was primed with an electric detonator and should have given a complete detonation. It is evident from the analysis that the explosion was not complete, perhaps because of a defective detonator. The partial detonation probably blew out of the hole more of the products than a simple burning caused by an igniter and guncotton would have done. This accounts for the difference in the analysis of 2150 and 2153. Thus the report on this one sample, apparently incorrect, tends to show the utility of this method of detection in actual working conditions, when a weak detonator causes trouble, as well as in the artificial method of burning the powder with an igniter and guncotton.

Samples 2140, 2143, and 2149 are not included in the analyses in Table 4. They were sent to the fuels laboratory for proximate analysis to see whether the determination of volatile matter would give any information as to the presence of explosive residues. The volatile matter in the samples was as follows[a] (moisture-free and ash-free basis).

Volatile matter in residues from three coals.

2140	39.09 per cent.
2143	40.55 per cent.
2149	39.75 per cent.

---

[a] Analyses by A. C. Fieldner, chemist, Bureau of Mines.

A slight increase in the volatile matter is noted in two samples of coal which contained explosive residue, but it is not believed that the difference is great enough to be of any assistance.

OBJECTIONS TO THE METHOD.

Objection has been raised to this method on two grounds. First, that the large particles caught on the 8-mesh sieve may contain some of the explosive residue. Second, that the use of a percentage basis is not best because the calculation does not consider that varying amounts of coal will be scraped from the holes, so that some samples are diluted more than others. The proposed remedy for this was to calculate the results obtained to the total weight of sample taken from each hole rather than to grams per 100 grams of 8-mesh sample. This would give the total weight of residue in each hole.

The reason for passing the samples through an 8-mesh sieve instead of grinding the entire sample was to get rid of the lumps of coal and thus enrich the sample. Dusting off the larger lumps with a camel's-hair brush, it was believed, would remove any explosive products adhering to them. To show that practically all of the residue was removed, the large particles from six samples after screening were ground in a mortar until they also passed the 8-mesh screen. Convenient quantities were taken and analyzed in the manner described. The only sample containing an appreciable percentage of soluble matter was one from the burning of a permissible explosive without detonation. In this the products of combustion had formed large lumps that remained on the screen and caused the soluble matter in the large particles to run even higher than in the small. The fact that not all of the explosive products pass through has no influence on the result. Enough was obtained through the screen to establish clearly that the explosive did not detonate and to identify the explosive used. Hence it is of no importance if more than half of the burned residue remains on the screen. In samples containing residue from black powder or detonated explosive the amount of explosives products in the large particles was negligible.

The second point, that calculation on a percentage basis is less accurate, is not sustained by close consideration. Obviously the same quantity of explosive is not used in every shot and a large quantity has a stronger tendency to blow out the solid products from the hole. On the other hand, its explosion would form more solid products of combustion. Whether the total quantity of residue found in the hole will vary directly with the quantity of explosive fired or inversely is uncertain. As this factor is apt to vary considerably it is thought that the calculation on a percentage basis is as good as the other, if the sampling is done in the manner described.

Attention is called to the fact that the method is not strictly quantitative. In the comments on the tables and in interpreting the results the semiquantitative results and the presence or absence of certain constituents were of much more value in identifying the samples than the precise analyses.

DIRECTIONS FOR SAMPLING.

The method of procedure if a bore hole suspected of having given a blown-out shot is found in a mine is as follows:

1. Obtain a sample of drillings from a hole in the same coal bed as that in which there were blown-out shots and as close as practicable to them.
2. Carefully remove any free powder or residue found in the hole of the blown-out shot, also any loose material around the collar. Then scrape or ream the hole through its entire length and combine all the residue and the scrapings from one hole into one sample, *totalling at least half a pound.*
3. Seal and carefully label the container.

If these directions for sampling are carefully followed, the authors believe that the method of examination and analysis herein described will give the desired information (p. 7).

SUMMARY.

1. Tests were made under known conditions to develop a method of detecting by chemical analysis the presence of residues of explosives in drill holes.
2. The method so developed was applied to a set of unknown samples with satisfactory results.
3. The success of the method depends considerably on the method of sampling.
4. From the analysis conclusions may be drawn regarding the nature of the explosive fired and method of firing.

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Bulletin 52. Ignition of mine gases by the filaments of incandescent electric lamps, by H. H. Clark and L. C. Ilsley. 1913. 31 pp., 6 pls., 2 figs.

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